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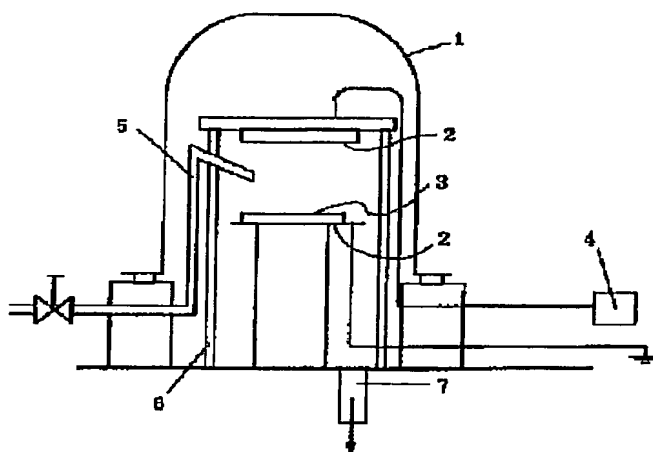
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APPLICANT : TOMOEGAWA PAPER CO LTD;

INVENTOR : INAGAKI KUNIHIRO;

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TITLE : HYDROPHILIC POROUS FLUORINE  
FIBER SHEET AND ITS PRODUCTION



ABSTRACT : PROBLEM TO BE SOLVED: To obtain a hydrophilic porous fluorine fiber sheet that is composed of a porous fluorine fiber sheet subjected to plasma treatment and then graft polymerization with a vinyl-based monomer having a sulfonic group, and useful as filter elements without discoloration and strength reduction.

SOLUTION: This hydrophilic porous fluorine fiber sheet is obtained by plasma-treating the surfaces of a porous fluorine fiber sheet 3 composed of tetrafluoroethylene resin or the like, and graft-polymerizing the resin with a vinyl-based monomer having a sulfonic group such as vinyl sulfonic group or vinylbenzene sulfonic group.

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## HYDROPHILIC POROUS FLUORINE FIBER SHEET AND ITS PRODUCTION

**Publication number:** JP9296368

**Publication date:** 1997-11-18

**Inventor:** TSUCHIDA MINORU; SUZUKI TAKANORI; KUROSAKI MASAARI; INAGAKI KUNIHIRO

**Applicant:** TOMOEGAWA PAPER CO LTD

**Classification:**

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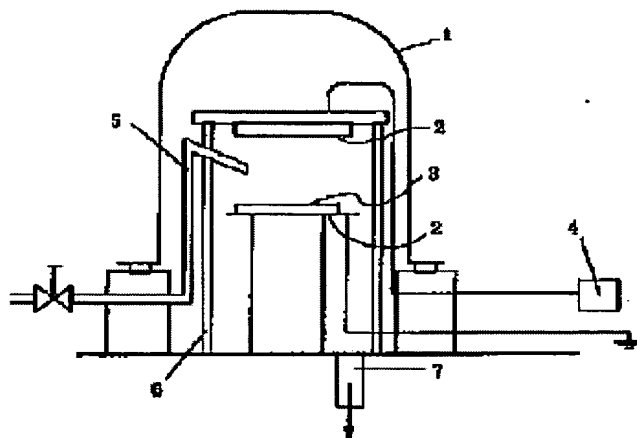
**Application number:** JP19960131338 19960425

**Priority number(s):** JP19960131338 19960425

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### Abstract of JP9296368

**PROBLEM TO BE SOLVED:** To obtain a hydrophilic porous fluorine fiber sheet that is composed of a porous fluorine fiber sheet subjected to plasma treatment and then graft polymerization with a vinyl-based monomer having a sulfonic group, and useful as filter elements without discoloration and strength reduction. **SOLUTION:** This hydrophilic porous fluorine fiber sheet is obtained by plasma-treating the surfaces of a porous fluorine fiber sheet 3 composed of tetrafluoroethylene resin or the like, and graft-polymerizing the resin with a vinyl-based monomer having a sulfonic group such as vinyl sulfonic group or vinylbenzene sulfonic group.



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**CLAIMS**

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[Claim(s)]

[Claim 1] The hydrophilic porosity fluorine fiber sheet characterized by the vinyl system monomer which has a sulfonic group on the front face of the porosity fluorine fiber sheet which performed plasma treatment coming to carry out graft polymerization.

[Claim 2] The manufacture approach of the hydrophilic porosity fluorine fiber sheet characterized by carrying out the graft polymerization of the vinyl system monomer which has a sulfonic group after performing plasma treatment to the front face of a porosity fluorine fiber sheet.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to a hydrophilic porosity fluorine fiber sheet useful as the various filter members and diffusion plate with which thermal resistance, chemical resistance, and dipping nature are demanded, an ion-exchange filter, the separator for cells, a catalyst, catalyst support, etc., and its manufacture approach.

[0002]

[Description of the Prior Art] Porosity fluorine fiber sheets, such as textile fabrics and a nonwoven fabric, are excellent in thermal resistance or chemical resistance, and since it is chemically stable, they are used for the application [ lower / exposure / of strong acid, a strong base and ultraviolet rays, or a radiation /, or oxidation reduction ambient atmosphere ] which is a filter member etc. under a severe condition chemically. However, since the front face is hydrophobicity, the conventional porosity fluorine fiber sheet has very low compatibility with water. Therefore, when wettability with water was dramatically bad and used it as a filter, there was a trouble that the dipping nature of a water solution was remarkable and it was bad. The hydrophilic grant to a porosity fluorine fiber sheet was difficult to carry out hydrophilization processing of the fluorine system macromolecule in the art by which the fluorine system macromolecule which constitutes a sheet is generally used for hydrophilization processing of a macromolecule since it is chemical very stable. On the other hand as the surface treatment approach of a fluorine system macromolecule, there is an approach using the liquid ammonia solution of metallic sodium, the complex compound solution which added and adjusted metallic sodium to the tetrahydrofuran solution of naphthalene, and the processing agent was also marketed. However, since these processing agents are powerful drugs, they are inferior to handling nature, and need waste fluid processing after processing. Furthermore, the treatment effect had a trouble of lowering of black change and a macromolecule on the strength for the front face of a fluorine system macromolecule.

[0003]

[Problem(s) to be Solved by the Invention] The object of this invention is with said conventional technique to offer the porosity fluorine sheet with which the impossible hydrophilic property was given, and its manufacture approach.

[0004]

[Means for Solving the Problem] It is what was made as a result of studying wholeheartedly the hydrophilization art which this invention solves the trouble of the commercial modifier of the above-mentioned fluorine system macromolecule, and does not cause lowering of a porosity fluorine fiber sheet on the strength, but can give a good hydrophilic property. By carrying out the polymerization of the vinyl system monomer which has a sulfonic group at least to the polymerization initiation section, after performing plasma treatment to a porosity fluorine fiber sheet and introducing at least the polymerization initiation section into the front face It found out that a good hydrophilic property

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could be given, without causing black change and lowering on the strength of a porosity fluorine fiber sheet. That is, it is the hydrophilic porosity fluorine fiber sheet characterized by the vinyl system monomer which has a sulfonic group on the front face of the porosity fluorine fiber sheet which performed plasma treatment coming to carry out the graft polymerization of the hydrophilic porosity fluorine fiber sheet of this invention, and the manufacture approach is the manufacture approach of the hydrophilic porosity fluorine fiber sheet characterized by to carry out the graft polymerization of the vinyl system monomer which has a sulfonic group, after performing plasma treatment to the front face of a porosity fluorine fiber sheet.

[0005] The hydrophilic porosity fluorine fiber sheet and the manufacture approach of this invention are explained in full detail. The porosity fluorine fiber sheet which constitutes this invention Tetrafluoroethylene resin, the tetrafluoroethylene perfluoro alkoxy ethylene copolymer resin, Tetrafluoroethylene and the 6 fluoride [ propylene ] copolymer resin, the tetrafluoroethylene ethylene copolymer resin, Polyvinylidene fluoride resin, 3 fluoride-salt-ized ethylene resin, the 3 fluoride-salt-ized ethylene ethylene copolymer resin, It consists of the textile fabrics or the nonwoven fabric of a fluorine system macromolecule which consists of kinds, such as tetrafluoroethylene-perfluoro dimethyl JIOKI SOL copolymer resin and polyvinyl fluoride, or two sorts or more, and what has good permeability is used. At least the polymerization initiation section is introduced into this porosity fluorine fiber sheet by performing plasma treatment with the following means beforehand. And the vinyl system monomer which has a sulfonic group comes to carry out graft polymerization at least to the polymerization initiation section. As a vinyl system monomer containing this sulfonic group, a vinyl sulfonic acid and its salt, a vinylbenzene sulfonic acid, its salt, etc. are mentioned. Moreover, if the vinyl fluorosulfonic acid which replaced a part or all of a hydrogen atom of these vinyl system monomer by the fluorine atom is used, chemical stability will improve further.

[0006] In manufacture of a porosity fluorine fiber sheet in which the vinyl system polymer which has the sulfonic group of this invention comes to carry out a graft, it is installation like the polymerization initiation section to (1) porosity fluorine fiber sheet front face.

(2) The polymerization of the vinyl system monomer in a porosity fluorine fiber sheet front face. \*\*\*\*\* is performed. Plasma treatment is carried out to installation like the polymerization initiation section in the front face of a porosity fluorine fiber sheet in which a vinyl system monomer and a reaction are possible. It can be used for the equipment used for plasma treatment, without limiting DC power supply, the power source of commercial frequency, the equipment that used microwave, or especially the equipment of an internal electrode method or an external electrode method. Moreover, the gas of non-polymerization nature, such as rare gas, such as helium and an argon, air, oxygen, nitrogen, and hydrogen, can be mixed in the plasma treatment gas used for plasma treatment, and a kind or two sorts or more can be used for it. A radical kind generates on the front face of a porosity fluorine fiber sheet by said plasma treatment, the pel oxide which this radical kind reacts with the oxygen in direct or atmospheric air, and produces reacts with the vinyl system monomer described below as a polymerization start point, and a polymerization advances.

[0007] The polymerization of the vinyl system monomer which has a sulfonic group is immersed in the vinyl system monomer which has a sulfonic group, or its solution, and carries out the polymerization of the porosity fluorine fiber sheet which carried out [ above-mentioned ] plasma treatment at suitable temperature. Moreover, as a solvent used for the solution of the vinyl system monomer which has a sulfonic group, if this monomer may be dissolved, it can use without a limit, for example, water, alcohols, and ketones will be used especially suitably.

[0008] Hereafter, this invention is explained based on an example.

The example 120cmx20cm porosity fluorine fiber sheet (TOMY fine lek R-250, nonwoven fabric made from : tetrafluoroethylene by Tomoegawa Paper Co., Ltd.) was applied to the internal electrode mold parallel plate plasma treatment equipment of drawing 1 , and plasma treatment of the front face of this sheet was carried out. The processing conditions in that case performed raw gas

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in argon gas, frequency [ of 13.56MHz ], output 10W, system internal pressure [ of 13.3Pa ], and processing-time 10 seconds. After exposing said porosity fluorine fiber sheet which carried out plasma treatment for 1 minute into atmospheric air, it was immersed in 100g of 10-% of the weight solutions of sodium vinylsulfonate, and polymerization was carried out at the temperature of 70 degrees C for 48 hours. Then, after the methanol washed, it was made to dry at the temperature of 50 degrees C for 12 hours, and the hydrophilic porosity fluorine fiber sheet of this invention was obtained.

[0009] Except using a vinylbenzene sulfonic-acid potassium for the vinyl system monomer which has example 2 sulfonic group, the same actuation as an example 1 was performed, and the hydrophilic porosity fluorine fiber sheet of this invention was obtained.

[0010] Without carrying out the graft polymerization by the vinyl system monomer which has plasma treatment and a sulfonic group to the porosity fluorine fiber sheet used in the example of comparison 1 example 1, using the commercial fluorine system giant-molecule modifier (trade name: fluoro dirty, NIPPON PILLAR PACKING CO., LTD. make), surface treatment was carried out and the hydrophilization porosity fluorine fiber sheet for a comparison was obtained.

[0011] The unsettled porosity fluorine fiber sheet for a comparison which does not perform the graft polymerization of the vinyl system monomer which has plasma treatment and a sulfonic group for the porosity fluorine fiber sheet used in the example of comparison 2 example 1 was obtained.

[0012] The porosity fluorine fiber sheet for a comparison was obtained like the example 1 except 10% water solution of sodium vinylsulfonate not performing a polymerization for the porosity fluorine fiber sheet used in the example of comparison 3 example 1.

[0013] The porosity fluorine fiber sheet for a comparison was obtained like the example 1 except not performing plasma treatment for the porosity fluorine fiber sheet used in the example of comparison 4 example 1.

[0014] The following item was examined about the porosity fluorine fiber sheet obtained in said example and the example of a comparison, and the result was shown in a table 1.

[0015] (1) Wettability test method : hang vertically the test piece of 25mm width of face, contact a soffit in water, and measure the height of the oil level which went up in 3 minutes.

[0016] (2) Tensile strength trial : JIS It was based on P8113.

[0017] (3) The color tone of a porosity fluorine fiber sheet was judged visually.

[0018]

[A table 1]

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試料	ぬれ性 (mm)	引っ張り強度 (kg / 15mm)	色調
実施例 1	90	0.6	白
実施例 2	90	0.6	白
比較例 1	70	0.2	黒
比較例 2	0	0.6	白
比較例 3	20	0.6	白
比較例 4	0	0.6	白

[0019] While examples 1 and 2 were excellent in the wettability which is the scale of a hydrophilic property, lowering of tensile strength and change of a color tone were not accepted so that clearly from the test result of a table 1. On the other hand, although wettability of the example 1 of a comparison improved, tensile strength fell and the color tone changed from white to black. Moreover, although the example 3 of a comparison had wettability slightly, it was checked that the examples 2 and 4 of a comparison do not have wettability.

[0020]

[Effect of the Invention] this invention -- change of the color tone of a porosity fluorine fiber sheet -- and -- \*\* -- the effectiveness which can give a good hydrophilic property to a sheet is done so, without causing lowering on the strength.

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**DESCRIPTION OF DRAWINGS**

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[Brief Description of the Drawings]

[Drawing 1] Drawing 1 is the sectional view of internal electrode mold parallel plate plasma treatment equipment.

[Description of Notations]

- 1 Bell Jar
- 2 Electrode
- 3 Sample
- 4 RF Generator
- 5 Raw Gas Supply Way
- 6 Electrode Stanchion
- 7 Exhaust Port

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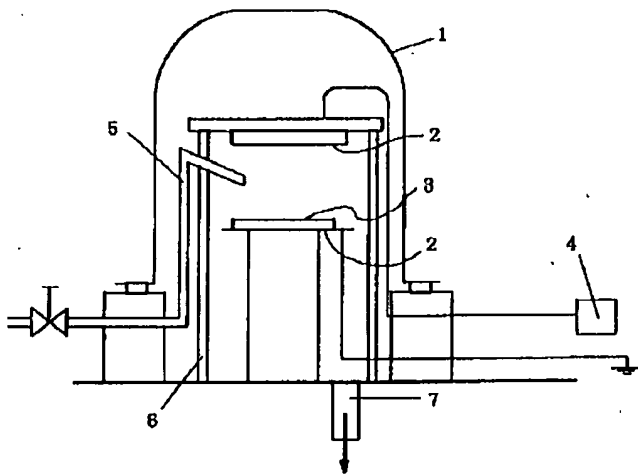
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**DRAWINGS**

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[Drawing 1]



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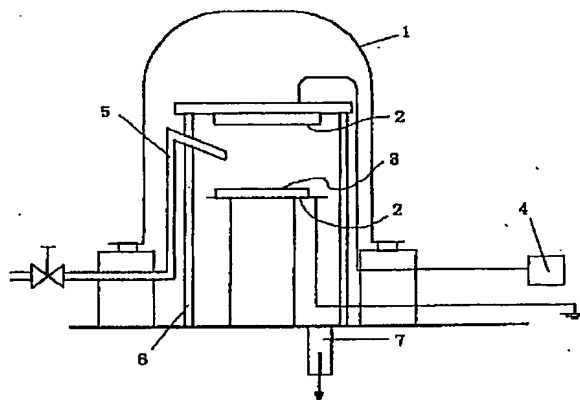
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(54)【発明の名称】 親水性多孔質フッ素繊維シート及びその製造方法

(57)【要約】

【目的】 本発明の目的は、多孔質フッ素繊維シートの色調変化及び強度低下の問題を問題が発生せず、親水性に優れた多孔質フッ素繊維シート及びその製造方法を提供すること。

【構成】 多孔質フッ素繊維シートをプラズマ処理し、スルホン酸基を有するビニル系単量体を重合した多孔質フッ素繊維シート及びその製造方法。



## 【特許請求の範囲】

【請求項1】 プラズマ処理を施した多孔質フッ素繊維シートの表面に、スルホン酸基を有するビニル系単量体がグラフト重合してなることを特徴とする親水性多孔質フッ素繊維シート。

【請求項2】 多孔質フッ素繊維シートの表面にプラズマ処理を施した後、スルホン酸基を有するビニル系単量体をグラフト重合させることを特徴とする親水性多孔質フッ素繊維シートの製造方法。

## 【発明の詳細な説明】

## 【0001】

【産業上の利用分野】本発明は、耐熱性、耐薬品性、通液性が要求される各種フィルター部材、散気板、イオン交換フィルター、電池用セパレータ、触媒及び触媒担体等として有用な親水性多孔質フッ素繊維シート及びその製造方法に関する。

## 【0002】

【従来の技術】織布、不織布等の多孔質フッ素繊維シートは、耐熱性や耐薬品性に優れ化学的に安定であるため、強酸や強塩基、紫外線や放射線の照射下、あるいは、酸化還元雰囲気下等での化学的に過酷な条件下でフィルター部材等の用途に用いられている。しかしながら、従来の多孔質フッ素繊維シートは、その表面が疎水性であるため、水との親和性が極めて低い。そのため、水とのぬれ性が非常に悪く、フィルタとして使用した場合は、水溶液の通液性が著しく悪いという問題点があった。多孔質フッ素繊維シートへの親水性付与は、シートを構成するフッ素系高分子が化学的に非常に安定であるため、一般に高分子の親水化処理に用いられる処理方法では、フッ素系高分子を親水化処理することが困難であった。一方、フッ素系高分子の表面改質方法としては、金属ナトリウムの液体アンモニア溶液や、金属ナトリウムをナフタリンのテトラヒドロフラン溶液に加えて調整した錯化合物溶液などを用いる方法があり、処理剤も市販されていた。しかし、これらの処理剤は、劇薬であるため、ハンドリング性に劣り、また、処理後に廃液処理が必要である。更に、その処理効果は、フッ素系高分子の表面を黒色変化、高分子の強度低下という問題点があった。

## 【0003】

【発明が解決しようとする課題】本発明の目的は、前記従来技術では不可能であった親水性が付与された多孔質フッ素シート及びその製造方法を提供することにある。

## 【0004】

【課題を解決するための手段】本発明は、上記フッ素系高分子の市販改質剤の問題点を解決し、多孔質フッ素繊維シートの強度低下を招かず、良好な親水性を付与できる親水化処理方法を鋭意研究した結果なされたもので、多孔質フッ素繊維シートにプラズマ処理を施し、その表面に重合開始部位を導入した後、その重合開始部位にス

ルホン酸基を有するビニル系単量体を重合させることにより、多孔質フッ素繊維シートの黒色変化及び強度低下を起こすことなく良好な親水性を付与できることを見いだした。すなわち、本発明の親水性多孔質フッ素繊維シートは、プラズマ処理を施した多孔質フッ素繊維シートの表面に、スルホン酸基を有するビニル系単量体がグラフト重合してなることを特徴とする親水性多孔質フッ素繊維シートであり、またその製造方法は多孔質フッ素繊維シートの表面にプラズマ処理を施した後、スルホン酸基を有するビニル系単量体をグラフト重合させることを特徴とする親水性多孔質フッ素繊維シートの製造方法である。

【0005】本発明の親水性多孔質フッ素繊維シート及び製造方法について詳述する。本発明を構成する多孔質フッ素繊維シートは、四フッ化エチレン樹脂、四フッ化エチレン・パーフルオロアルコキシエチレン共重合体樹脂、四フッ化エチレン・六フッ化プロピレン共重合体樹脂、四フッ化エチレン・エチレン共重合体樹脂、フッ化ビニリデン樹脂、三フッ化塩化エチレン樹脂、三フッ化塩化エチレン・エチレン共重合体樹脂、テトラフルオロエチレンーパーフルオロジメチルジエチレン共重合体樹脂、フッ化ビニル樹脂等の一種あるいは二種以上からなるフッ素系高分子の織布あるいは不織布からなり、良好な通気性を有するものが用いられる。該多孔質フッ素繊維シートには、予め下記的手段によってプラズマ処理を施すことにより重合開始部位が導入される。そして重合開始部位にスルホン酸基を有するビニル系単量体がグラフト重合してなったものである。該スルホン酸基を含有するビニル系単量体としては、ビニルスルホン酸及びその塩やビニルベンゼンスルホン酸及びその塩などが挙げられる。また、これらビニル系単量体の水素原子の一部あるいは全部をフッ素原子で置き換えたビニルフルオロスルホン酸類を用いると、化学的な安定性が更に向上する。

【0006】本発明のスルホン酸基を有するビニル系重合体がグラフトしてなる多孔質フッ素繊維シートの製造には、

(1)多孔質フッ素繊維シート表面への重合開始部位の導入。

(2)多孔質フッ素繊維シート表面におけるビニル系単量体の重合。

の工程が行われる。多孔質フッ素繊維シートの表面におけるビニル系単量体と反応可能な重合開始部位の導入には、プラズマ処理が行われる。プラズマ処理に用いる装置には、直流電源や商用周波数の電源、マイクロ波を用いた装置、あるいは、内部電極方式や外部電極方式の装置などを特に限定することなく使用することができる。また、プラズマ処理に用いるプラズマ処理ガスには、ヘリウムやアルゴンなどの希ガス、空気、酸素、窒素、水素等の非重合性のガスを一種あるいは二種以上を混合し

て用いることができる。前記プラズマ処理により多孔質フッ素繊維シートの表面にラジカル種が生成し、このラジカル種が、直接あるいは、大気中の酸素と反応して生じるペルオキサイドが重合開始点として以下に述べるビニル系単量体と反応し、重合が進行する。

【0007】スルホン酸基を有するビニル系単量体の重合処理は、上記プラズマ処理した多孔質フッ素繊維シートをスルホン酸基を有するビニル系単量体あるいはその溶液に浸漬し、適切な温度で重合させる。また、スルホン酸基を有するビニル系単量体の溶液に用いる溶媒としては、この単量体を溶解しうるものならば特に制限無く用いることが出来、例えば、水、アルコール類、ケトン類等が好適に使用される。

【0008】以下、実施例に基づき本発明を説明する。

#### 実施例1

20cm×20cmの多孔質フッ素繊維シート（トミーファインレック R-250、巴川製紙所社製：四フッ化エチレン製不織布）を図1の内部電極型平行平板プラズマ処理装置に適用し、該シートの表面をプラズマ処理した。その際の処理条件は、処理ガスをアルゴンガス、周波数13.56MHz、出力10W、系内圧力13.3Pa、処理時間10秒で行った。前記プラズマ処理した多孔質フッ素繊維シートを大気中に1分間暴露した後、ビニルスルホン酸ナトリウムの10重量%溶液100gに浸漬し、70℃の温度で48時間重合処理した。その後、メタノールで洗浄した後、50℃の温度で12時間乾燥させ、本発明の親水性多孔質フッ素繊維シートを得た。

#### 【0009】実施例2

スルホン酸基を有するビニル系単量体にビニルベンゼン  
スルホン酸カリウムを使用する以外は、実施例1と同様な操作を行い本発明の親水性多孔質フッ素繊維シートを得た。

#### 【0010】比較例1

実施例1で使用した多孔質フッ素繊維シートに対し、プラズマ処理及びスルホン酸基を有するビニル系単量体によるグラフト重合をすることなく、市販のフッ素系高分子改質剤（商品名：フルオロエッチ、日本ピラー工業社製）を用いて、表面処理し、比較用の親水化多孔質フッ素繊維シートを得た。

#### 【0011】比較例2

実施例1で使用した多孔質フッ素繊維シートをプラズマ処理及びスルホン酸基を有するビニル系単量体のグラフト重合を行わない比較用の未処理の多孔質フッ素繊維シートを得た。

#### 【0012】比較例3

実施例1で使用した多孔質フッ素繊維シートをビニルスルホン酸ナトリウムの10%水溶液にて重合を行わない以外は実施例1と同様にして比較用の多孔質フッ素繊維シートを得た。

#### 【0013】比較例4

実施例1で使用した多孔質フッ素繊維シートをプラズマ処理を行わない以外は実施例1と同様にして比較用の多孔質フッ素繊維シートを得た。

【0014】前記実施例及び比較例で得られた多孔質フッ素繊維シートについて下記の項目の試験を行い、結果を表1に示した。

【0015】（1）ぬれ性試験方法：25mm幅の試料片を垂直に吊り下げ、下端を水に接触させて、3分間で上昇した液面の高さを測定する。

【0016】（2）引っ張り強度試験：JIS P8113に準拠した。

【0017】（3）多孔質フッ素繊維シートの色調を目視にて判断した。

【0018】

【表1】

試料	ぬれ性 (mm)	引っ張り強度 (kg/15mm)	色調
実施例 1	90	0.6	白
実施例 2	90	0.6	白
比較例 1	70	0.2	黒
比較例 2	0	0.6	白
比較例 3	20	0.6	白
比較例 4	0	0.6	白

【0019】表1の試験結果から明らかなように実施例1及び2は親水性の尺度であるぬれ性に優れると同時に引っ張り強度の低下及び色調の変化は認められなかった。これに対して、比較例1はぬれ性は向上するが、引っ張り強度が低下し色調が白から黒色へ変化した。また比較例3は僅かにぬれ性はあるが、比較例2、4はぬれ性がないことが確認された。

【0020】

【発明の効果】本発明は、多孔質フッ素繊維シートの色調の変化及び強度低下を起こすことなく、シートに良好な親水性を付与できる効果を奏する。

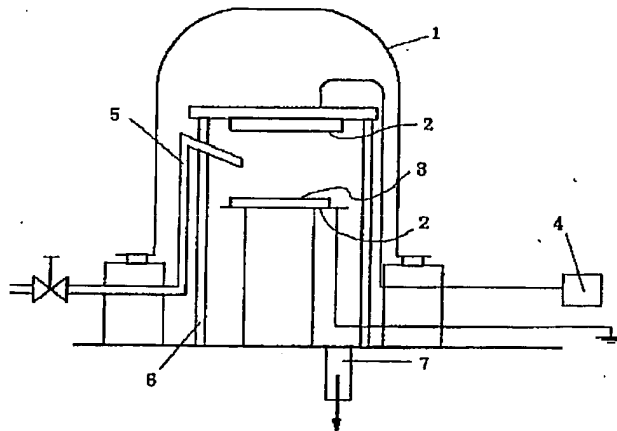
\* 【図面の簡単な説明】

20 【図1】図1は内部電極型平行平板プラズマ処理装置の断面図である。

【符号の説明】

- 1 ベルジャー
- 2 電極
- 3 試料
- 4 高周波電源
- 5 処理ガス供給路
- 6 電極支柱
- 7 排気口

【図1】



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